

# CARBON FIBER REINFORCED CEMENT IMPROVED BY USING SILANE TREATED CARBON FIBERS

*Yunsheng Xu and D.D.L. Chung*  
*Composite Materials Research Laboratory*  
*State University of New York at Buffalo*  
*Buffalo, NY 14260-4400, USA*

## Introduction

Cement reinforced with short carbon fibers is attractive due to its high flexural strength and toughness and low drying shrinkage, in addition to its strain sensing ability [1-12]. Surface treatment of carbon fibers by ozone has been shown to be effective for improving the wettability by water, thereby improving the fiber-matrix bond, increasing the tensile strength, modulus and ductility beyond the levels attained with untreated carbon fibers, and decreasing the drying shrinkage below the levels attained with untreated fibers [13]. We have recently reported that the surface treatment of silica fume by using a silane coupling agent improves both workability and strengths (tensile and compressive) of cement mortar beyond the levels attained by using untreated silica fume, due to the hydrophylic nature of the silane molecule and the consequent improved wettability of silica fume by water [14]. In this paper, we report that the silane treatment conducted on carbon fibers improves the mechanical properties of carbon fiber reinforced cement paste beyond the levels attained by using ozone treated carbon fibers.

Silica fume is used along with carbon fibers in order to help the dispersion of the fibers in the cement mix [5]. In this paper, we also report that the combined use of silane treated carbon fibers and silane treated silica fume results in mechanical properties that are superior to those attained by silane treated carbon fibers in combination with untreated silica fume or those attained by untreated carbon fibers in combination with silane treated silica fume.

## Experimental Methods

The carbon fibers were isotropic pitch based, unsized, and of length ~ 5 mm, as obtained from Ashland Petroleum Co. (Ashland, Kentucky). As-received and three types of surface treated fibers were used. The fiber content was 0.5% by weight of cement. The surface treatments involved (i) ozone ( $O_3$ ), (ii) an aqueous solution of potassium dichromate ( $K_2Cr_2O_7$ , 30 wt.%) and sulfuric acid ( $H_2SO_4$ , 40 wt.%, which enhances the oxidation ability) and (iii) silane. The ozone treatment for surface

oxidation involved exposure of the fibers to  $O_3$  gas (0.6 vol.%, in  $O_2$ ) at  $160^\circ C$  for 5 min. Prior to  $O_3$  exposure, the fibers had been dried at  $160^\circ C$  in air for 30 min. The potassium dichromate treatment for surface oxidation involved immersing in the dichromate solution and heating to  $60^\circ C$  while stirring for 2 h, followed by filtration and washing with water and then drying at  $110^\circ C$  for 6 h. For the silane treatment, the silane coupling agent was a 1:1 (by weight) mixture of Z-6020 ( $H_2NCH_2CH_2NHCH_2CH_2CH_2Si(OCH_3)_3$ ) and Z-6040 ( $(OCH_2CH_2CH_2OCH_2CH_2CH_2Si(OCH_3)_3)$ ) from Dow Corning Corp. (Midland, MI). The amine group in Z-6020 serves as the catalyst for the curing of epoxy and consequently allows the Z-6020 molecule to attach to the epoxy end of the Z-6040 molecule. The trimethylsiloxy ends of the Z-6020 and Z-6040 molecules then connect to the -OH functional group on the surface of silica fume or carbon fibers. The silane was dissolved in ethylacetate. Surface treatment was performed by immersing in the silane solution, heating to  $75^\circ C$  while stirring, and holding at  $75^\circ C$  for 1 h, followed by filtration, washing with ethylacetate, and drying. After this, heating was conducted in a furnace at  $110^\circ C$  for 12 h.

No aggregate (fine or coarse) was used. The water/cement ratio was 0.35. A water-reducing agent (TAMOL SN, Rohm and Haas Co., Philadelphia, PA; sodium salt of a condensed naphthalenesulphonic acid) was used in the amount of 2% by weight of cement.

The cement used was portland cement (Type I) from Lafarge Corp. (Southfield, MI). The silica fume (Elkem Materials, Inc., Pittsburgh, PA, EMS 965) was used in the amount of 15% by weight of cement. The methylcellulose, used in the amount of 0.4% by weight of cement, was Dow Chemical, Midland, MI, Methocel A15-LV. The defoamer (Colloids Inc., Marietta, GA, 1010) used whenever methylcellulose was used in the amount of 0.13 vol.%.

A rotary mixer with a flat beater was used. Methylcellulose (if applicable) was dissolved in water and then the defoamer was added and stirred by hand for about 2 min. Then this mixture (if applicable), cement, water, water reducing agent, silica fume and fibers (if applicable)

were mixed in the mixer for 10 min. After pouring into molds, an external vibrator was used to facilitate compaction and decrease the amount of air bubbles. The samples were demolded after 24 h and then cured in air at room temperature and a relative humidity of 100% for 28 days.

## Results

The tensile strength is slightly increased by the addition of methylcellulose and defoamer, but the modulus is slightly decreased by the addition of methylcellulose and defoamer. However, both strength and modulus are increased by the addition of fibers. The effectiveness of the fibers in increasing strength and modulus increases in the order: as-received fibers, O<sub>3</sub>-treated fibers, dichromate-treated fibers and silane-treated fibers. This trend applies whether the silica fume is as-received or silane-treated. For any of the formulations, silane-treated silica fume gives substantially higher strength and modulus than as-received silica fume. The highest tensile strength and modulus are exhibited by cement paste with silane-treated silica fume and silane-treated fibers. The strength is 56% higher and the modulus is 39% higher than those of the cement paste with as-received silica fume and as-received fibers. The strength is 26% higher and the modulus is 14% higher than those of the cement paste with as-received silica fume and silane-treated fibers. Hence, silane treatments of silica fume and of fibers are about equally valuable in providing strengthening.

The air void content is decreased by the addition of methylcellulose and defoamer, but is increased by the further addition of fibers, whether the fibers have been surface treated or not. Among the formulations with fibers, the air void content decreases in the order: as-received fibers, O<sub>3</sub>-treated fibers, dichromate-treated fibers and silane-treated fibers. This trend applies whether the silica fume is as-received or silane-treated. For any of the formulations (including those without fibers), silane-treated silica fume gives lower air void content than as-received silica fume.

## Conclusion

The tensile strength, modulus and ductility were increased and the air void content was decreased, when the fibers in carbon fiber reinforced cement paste had been surface treated. The effectiveness of treatment decreased in the order: silane, dichromate and ozone. Additional strengthening and air void content reduction were observed when the silica fume in the carbon fiber reinforced cement paste had been surface treated with silane. These effects of silane treatment are attributed to the hydrophylic nature of silane.

## References

- [1] Chen PW, Chung DDL. Low-drying-shrinkage concrete containing carbon fibers. *Composites* 1996;Part B, 27B:269-274.
- [2] Chen PW, Chung DDL. A comparative study of concretes reinforced with carbon, polyethylene and steel fibers and their improvement by latex addition. *ACI Materials J* 1996;93(2):129-133.
- [3] Chen PW, Chung DDL. Concrete as a new strain/stress sensor. *Composites* 1996;Part B 27B:11-23.
- [4] Fu X, Chung DDL. Self-monitoring of fatigue damage in carbon fiber reinforced cement. *Cem Concr Res* 1996;26(1):15-20.
- [5] Chen PW, Fu X, Chung DDL. Microstructural and mechanical effects of latex, methylcellulose and silica fume on carbon fiber reinforced cement. *ACI Mater J* 1997;94(2):147-155.
- [6] Hou J, Chung DDL. Cathodic protection of steel reinforced concrete facilitated by using carbon fiber reinforced mortar or concrete. *Cem Concr Res* 1997;27(5):49-656.
- [7] Fu X, Ma E, Chung DDL, Anderson WA. Self-monitoring in carbon fiber reinforced mortar by reactance measurement. *Cem Concr Res* 1997;27(6):845-852.
- [8] Shi ZQ, Chung DDL. Improving the abrasion resistance of mortar by adding latex and carbon fibers. *Cem Concr Res* 1997;27(8):1149-1153.
- [9] Bantia N. Carbon fiber cements: structure, performance, applications and research needs. *ACI SP-142, Fiber Reinforced Concrete*, J.I. Daniel and S.P. Shah, Ed., ACI, Detroit, MI, 1994, p. 91-119.
- [10] Toutanji HA, El-Korchi T, Katz RN. Strength and reliability of carbon-fiber-reinforced cement composites. *Cem Concr Composites* 1994;16:15-21.
- [11] Soroushian P, Nagi M, Hsu J. Optimization of the use of lightweight aggregates in carbon fiber reinforced cement. *ACI Mater J* 1992;89(3):267-276.
- [12] Sakai H, Takahashi K, Mitsui Y, Ando T, Awata M, Hoshijima T. Flexural behavior of carbon fiber reinforced cement composite. *ACI SP-142, Fiber Reinforced Concrete*, J.I. Daniel and S.P. Shah, Ed., ACI, Detroit, MI, 1994, p. 121-140.
- [13] Fu X, Lu W, Chung DDL. Ozone treatment of carbon fiber for reinforcing cement. *Carbon* 1998;36(9):1337-1345.
- [14] Xu Y, Chung DDL. Improving the workability and strength of silica fume concrete by using silane treated silica fume. *Cem Concr Res.*, in press.